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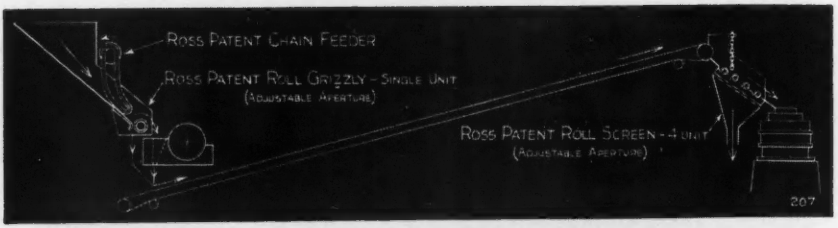

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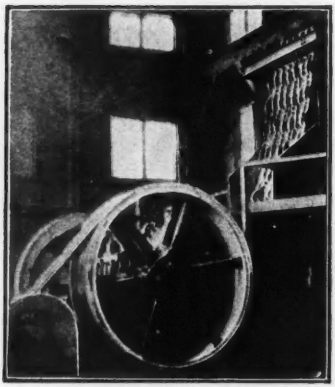

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PUBLISHED BY
CONCRETE PUBLICATIONS LIMITED
14 DARTMOUTH STREET, LONDON, S.W.1

TELEPHONE: WHITEHALL 4581.
TELEGRAPHIC ADDRESS:
CONCRETIUS, PARL, LONDON

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VOLUME XIV. NUMBER 11 NOVEMBER 1941

Specification for Measuring the Fineness of Portland Cement.

THE following is a revised "Tentative Method of Test for Fineness of Portland Cement by means of the Turbidimeter" which has been submitted to the American Society for Testing Materials.

(1) *Scope.*—This method of test covers the Wagner turbidimeter apparatus and procedure for determining the fineness of Portland cement as represented by specific surface expressed as total surface area in square centimetres per gram of cement.

(2) *Apparatus.*—The Wagner turbidimeter consists essentially of a source of light of constant intensity adjusted so that approximately parallel rays of light pass through a suspension of the cement to be tested and impinge upon the sensitive plate of a photoelectric cell. The current generated by the cell is measured with a microammeter and the indicated reading is a measure of the turbidity of the suspension. General considerations indicate that turbidity is in turn a measure of the surface area of the suspended sample of cement. The apparatus, illustrated in *Fig.* 1, shall consist specially of the parts described in Sections 3 to 9.

(3) *Turbidimeter.*—The turbidimeter shall be mounted in a suitable case and shall include the following features:

(*a*) *Source of Light.*—The source of light (*Fig.* 1) shall consist of a 3 to 6 candle-power concentrated-filament electric lamp operated by a 6-volt storage battery, or a source of constant electro-motive force. A parabolic reflector shall be mounted behind the lamp and the lamp focused in such manner that approximately parallel rays of light will pass through the sedimentation tank and will impinge

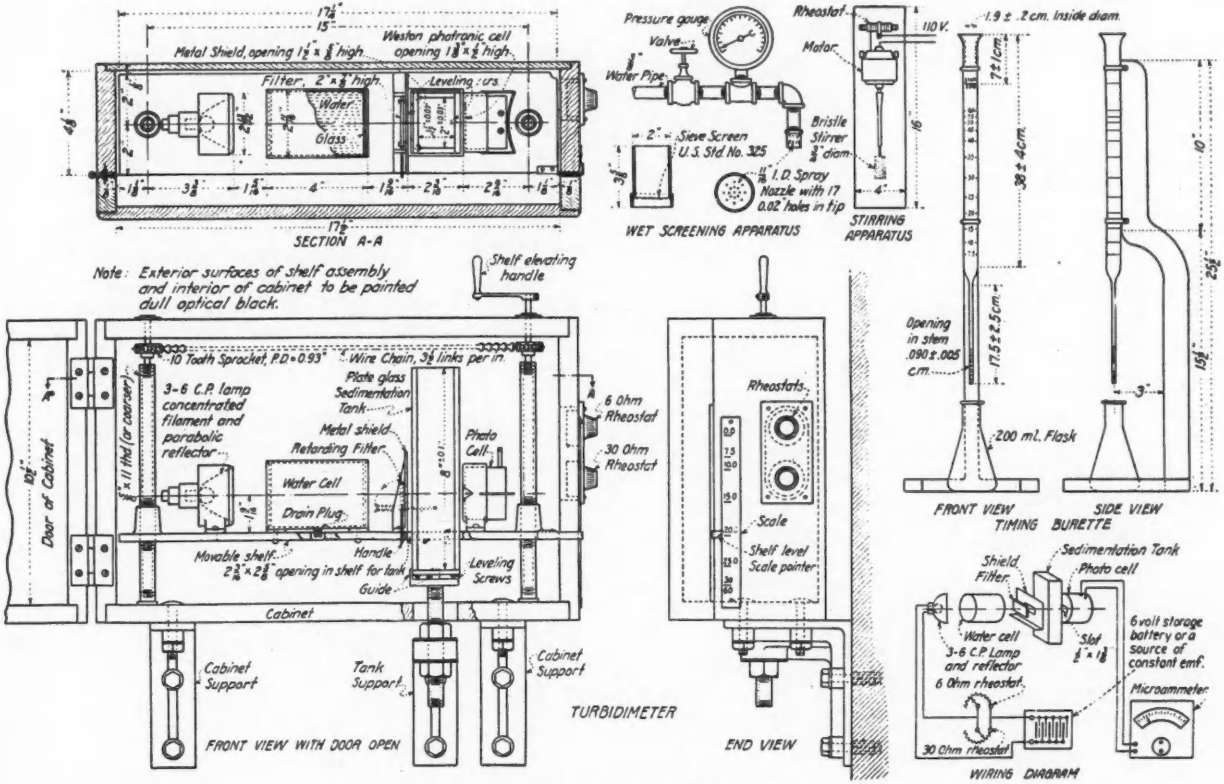

Fig. 1.—Details of Wagner Turbidimeter.

upon the photoelectric cell. The light intensity shall be regulated by means of two rheostats having resistances of approximately 6 and 30 ohms respectively, and mounted in series with the lamp but in parallel with each other.

(*b*) *Water Cell.*—The light shall pass through a water cell before entering the sedimentation tank in order that radiant heat from the beam shall be absorbed. The cell shall consist of 3 in. diameter brass tubing, 4 in. in length with glass windows cemented in the ends with a suitable sealing material. A stoppered hole shall provide for filling with distilled water. The cell shall be so arranged that all rays of light entering the sedimentation tank shall first pass through the water.

(*c*) *Retarding Filter.*—A light-retarding glass or other device shall be provided which will reduce the intensity of light from that corresponding to 100 microamperes to 20 to 30 microamperes. The light intensity shall be uniformly retarded over the entire area of that portion of the cell which is exposed to light during a test.

(*d*) *Sedimentation Tank.*—The sedimentation tank shall be constructed of $\frac{3}{16}$ in. plate glass cemented together to form a rectangular tank having inside dimensions 2 in. by $1\frac{1}{2}$ in. by 8 in. in height. The permissible variations of the inside dimensions of the tank shall be plus or minus 0·03 in. in length and width and plus or minus 0·1 in. in height. The 2 in. faces of the tank shall be placed normal to the beam of light, and shall be equidistant within 0·01 in. at all points. A mark shall be placed on the side of the tank to indicate a volumetric content of 335 ml., which is the level to which the tank will be filled in a test.

(*e*) *Photoelectric Cell.*—The means of measuring the light intensity shall be a sensitive photoelectric cell (Weston Photronic type) connected directly to a microammeter. A hood with a horizontal slot $\frac{1}{2}$ in. in height by $1\frac{3}{8}$ in. in width shall be mounted over the face of the photoelectric cell.

(*f*) *Shield.*—A metallic shield having a slot $\frac{5}{8}$ in. in height by $1\frac{1}{2}$ in. in width, as indicated in *Fig.* 1, shall be placed between the water cell and the sedimentation tank.

(*g*) *Elevating Device.*—The source of light, water cell, photoelectric cell, retarding filter, and shield shall be mounted on a movable shelf which may be raised or lowered and which may be readily and accurately adjusted so that the turbidity of the suspension may be determined at any desired depth. The sedimentation tank shall be mounted on a base which is independent of the rest of the apparatus so that the tank shall be free from vibration caused by moving the shelf. The level of the light beam with reference to the surface of the suspension shall be indicated by a pointer which will travel along a scale mounted on the cabinet. The zero of the scale shall indicate that position at which the centre lines of the slots for the light beam are at the same elevation as the surface of the liquid in the tank when filled to the 335-ml. level. The lines on the scale to be marked 7·5, 10, 15, 20, 25, and 30-60, shall be at distances from the zero mark equal to values *h* in *Table* I, and shall indicate the positions at which

TABLE I.—VALUES OF h, d, AND $\frac{h}{d^2}$ TO BE USED IN CALIBRATION OF THE TURBIDIMETER APPARATUS

Particle diameter, d, microns	Depth of Suspension, h, cm.	$\frac{h}{d^2}$
60	15	0·00417
55	15	0·00496
50	15	0·00600
45	15	0·00741
40	15	0·00938
35	15	0·01225
30	15	0·01667
25	13·1	0·0210
20	10·1	0·0250
15	6·6	0·0293
10	3·3	0·0333
7·5	2·1	0·0375

the pointer should be located when turbidity readings for these particle sizes are taken. The interior of the turbidimeter cabinet and the exterior surfaces of the shelf, parabolic reflector, water cell, shield, and photoelectric cell hood shall be painted with a dull optical black paint.

(4) *Microammeter.*—The microammeter shall have a range of 0 to 50 microamperes, shall be readable to 0·1 microampere, and accurate to 0·25 microampere. The internal resistance of the microammeter shall be between 50 and 150 ohms.

(5) *Source of Current.*—A 6-volt automobile starting and lighting storage battery or a source of constant e.m.f. shall be used for supplying current to the lamp.

(6) *Sieve.*—The sieve shall be 2 in. in diameter and have side walls 3 in. in height, and shall be fitted with a No. 325 (44 micron) woven wire screen cloth which shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation E 11) of the American Society for Testing Materials, and be such that the sieve correction factor obtained shall not be greater than 15 per cent.

(7) *Stirring Apparatus.*—The stirring apparatus shall consist of either (*a*) a cylindrical brush $\frac{3}{4}$ in. in diameter and about $1\frac{3}{4}$ in. in length, with an end approximately fitting the contour of the bottom of a $\frac{7}{8}$-in. diameter test tube or (*b*) any other stirring device which will be equally efficient in dispersion as measured by specific surface determinations on a standard sample. The stirring apparatus shall rotate at a speed of approximately 3,500 r.p.m.

(8) *Timing Burette.*—The time of settling for the different sized particles shall be obtained from a burette from which kerosene is allowed to flow. The burette shall consist of a glass tube having a capillary tube fused into the lower end. The upper end of the large tube shall be flared to serve as a funnel for introducing kerosene into the tube. The burette shall conform to the following limiting dimensions.

	Dimension, cm.	Permissible Variation, (plus or minus) cm.
Length of large tube	38	4
Inside diameter of large tube ..	1.9	0·2
Length of capillary	17·5	2·5
Diameter of capillary	0·09	0·005
Top of burette to zero line ..	7	1

Since glass tubing of desired dimensions is not always obtainable, the above wide permissible variations allow selection of dimensions to produce a burette having a duration of flow which will permit calibration.

(9) *Suspending Liquid.*—Clear white kerosene shall be used with the turbidimeter apparatus.

(*Sections* 10 *and* 11 *deal with the calibration of the turbidimeter and the size of test samples.*)

(12) *Sieve Determination.*—A No. 325 (44-micron) sieve determination shall be made on a 1-g. sample of cement. The percentage (corrected) of cement passing the sieve shall be recorded as *r*. After three determinations the sieve should be dipped in diluted HCl (1 : 10) and immediately rinsed with water to remove particles lodged in the meshes. After 25 determinations the sieve should be recalibrated. (See *Table* II, footnote *a*.)

(13) *Preparation of the Suspension.*—(*a*) The cement sample shall be placed in a test tube with 10 to 15 ml. of kerosene and 5 drops of oleic acid (U.S.P.). (Oleic acid is added as a dispensing agent and has slight or no effect on the viscosity of the kerosene. The oleic acid, which changes with age and exposure to heat and light, should be kept in a well-stoppered dark-glass bottle.) The mixture shall then be stirred for 1 minute with the apparatus described in Section 7 and then be transferred to the settling tank, the brush and tube washed with clear kerosene which shall also be added to the suspension, and clear kerosene added until the total volume of the suspension in the tank is 335 ml. Since it is important that the level of the liquid in the tank coincide with the zero mark on the scale of the instrument [see Section 3 (*g*)], it is correspondingly important that the volume of the suspension may be close to 335 ml. This may be accomplished by the use of a flask, calibrated to deliver 335 ml., from which all the kerosene used in making up a suspension is taken ; or a point gauge may be used, consisting essentially of a flat plate approximately 2 in. by 2½ in. to the centre of which is attached vertically a metal pin of such length that, when the plate is placed in position on top of the tank, the point of the pin is at the 335-ml. level.

(*b*) *Agitation of the Suspension.*—Just before being placed in the path of the light beam, the tank shall be agitated to effect a uniformity of the suspension. The tank shall be covered with a ground-glass cover and oscillated 180 deg. about a horizontal axis, turning the tank upside down and back again to its original upright position for about one minute. Precautions should be taken to prevent

any loss of the kerosene. The suspension will then be ready to place in its proper position in the path of the light beam. The procedure described for oscillating the suspension must be carefully followed. The faces of the glass tank shall be clean when the suspension is added, and care must be taken to avoid as much as possible the necessity of cleaning the faces thereafter.

TABLE II.—ILLUSTRATIVE FORM FOR RECORDING TURBIDIMETER TEST DATA AND CALCULATION OF SPECIFIC SURFACE

Sample identification	Sample X
Passing No. 325 (44-micron) sieve (wet method), r, corrected	89·7 per cent. [a]
Filter reference (through filter alone)	
Before test	20·5 microamperes
After test	20·5 microamperes
Weight of sample tested	0·3 g.

Particle Size, microns	I, microamperes		Log I
60	11·0	....	1·041 [b]
55	11·1	1·045	
50	11·3	1·053	
45	11·5	1·061	
40	11·7	1·068	
35	12·1	1·083	
30	12·6	1·100	
25	13·4	1·127	
20	14·4	1·158	
15	15·7	1·196	
10	19·1	1·281	
7·5	23·0	....	1·362 [b]
	0·75 × 1·362 =	1·022	
		1·500	
	Sum =	13·694	
	11·5 × 1·041 =	11·972	
	Difference =	1·722	

$$S = \frac{38 \times 89{\cdot}7 \times 0{\cdot}959}{1{\cdot}722} = 1900 \text{ sq. cm. per g.}$$

---

[a] The following example presents an illustration of the determination and application of the sieve correction factor for a No. 325 sieve (wet method):

Determination:

Residue specified for standard sample No. 114c = 10·8 per cent., or a residue for 1-g. test sample = 0·108 g.
Residue obtained on the sieve, which is being calibrated with standard sample = 0·100 g.

Difference between specified and test residues = 0·008 g.

Correction factor for sieve (plus in this example) $= \frac{+\ 0{\cdot}008}{0{\cdot}100} \times 100 = +\ 8{\cdot}0$ per cent.

Application:

Sieve correction factor = + 8·0 per cent.
Residue from the sample under test = 0·095 g.
Correction = + 0·08 × 0·095 g. = + 0·008 g.

Corrected residue (algebraic sum) = 0·103 g. = 10·3 per cent.
Corrected amount passing sieve (r) = 89·7 per cent.

[b] For convenience in calculation, log I_{60} and log $I_{7{\cdot}5}$ are recorded in a separate column.

(14) *Procedure.*—The turbidimeter apparatus shall be operated in accordance with the procedure described in the following paragraphs (*a*) to (*h*) :

(*a*) With retarding filter and sedimentation tank containing approximately 100 ml. of clear kerosene in the light path, the light shall be adjusted to the proper intensity, I_0, by taking repeated readings at 1-minute intervals until an unchanging value shall indicate that the lamp and photoelectric cell are in equilibrium.

(*b*) The proper size of sample of cement shall be weighed and suspension prepared in accordance with Section 13.

(*c*) The shelf level pointer shall be placed at the 30-60 micron position.

(*d*) The burette shall be filled to a suitable height with kerosene from the same lot and at the same temperature as the kerosene used in the suspension, and oscillation of the tank containing the suspension shall be started in accordance with Section 13 (*b*). The oscillation shall be continued until the kerosene drains to the zero line on the burette, when the agitation shall be stopped. The lid to the tank must fit so that no kerosene drains down the outside faces of the tank. The tank shall be immediately placed in position in the path of the light beam.

(*e*) The retarding filter shall be immediately removed from the light path and the cabinet door closed.

(*f*) The microammeter shall be read at the instant the kerosene in the burette drains past marks 60, 55, 50, 45, 40, 35, and 30.

(*g*) The shelf shall then be raised successively to the marks 25, 20, 15, 10, and 7·5 on the pointer scale, the microammeter being read at each position as the kerosene drains past the corresponding mark on the burette.

(*h*) The filter shall be replaced in the path of the light beam, the tank removed, and the intensity of the lamp checked. If the microammeter indication has shifted more than 0·3 microampere from the initial setting through the filter alone, the test must be repeated. A freshly-charged storage battery should be momentarily short-circuited to reduce the voltage to constant value.

(15) *Calculation and Record.*—(*a*) *Specific Surface.*—Specific surface shall be calculated from the following equation :

$$S = \frac{38r\,(2 - \log I_{60})}{1{\cdot}5 + 0{\cdot}75\,I_{7{\cdot}5} + \log I_{10} + \log I_{15} + \log I_{20} \ldots + \log I_{55} - 11{\cdot}5 \log I_{60}}$$

where

S = Specific surface of the sample in square centimetre per gram,

r = corrected percentage by weight passing the No. 325 (44-micron) sieve, and

$I_{7{\cdot}5}$, I_{10}, $I_{15} \ldots I_{60}$ = microammeter readings, in microamperes, which correspond to the particle diameters 7·5, 10, 15 .. 60 microns.

The constant factor 38 applies only to a material having the specific gravity of Portland cement (approximately 3·15).

(*b*) *Recording of Data.*—The form shown in *Table* II is suggested for the recording of turbidimeter data and calculation of specific surface.

(16) *Calculation of Specific Surface from First Turbidity Reading* I_{60}.—(*a*) This method may be used for successive determinations from the same plant, provided that the same size test samples are used and that there is no great change in fineness, colour, or other properties of the cement. Under these conditions the specific surface of a sample may be calculated from the first turbidity reading, I_{60}, by using the following equation :

$$S = c(2 - \log I_{60})$$

where

S = specific surface of the sample in square centimetres per gram,

I_{60}= microammeter reading corresponding to particle diameter of 60 microns, and

c . .= transmittancy constant for the particular plant.

The transmittancy constant c may be evaluated by substituting in the equation known values of S and $2-\log I_{60}$ as determined from a complete turbidimeter test. The average of not less than five values of c, as determined from complete tests, should be used in the above equation.

(*b*) If the fineness of a sample as determined in accordance with paragraph (*a*) fails to conform to the specification requirements for fineness, a retest shall be made by the complete procedure described in Sections 14 and 15.

B.S.S. for Portland Blastfurnace Cement.

A REVISION (dated September, 1941) of the British Standard Specification for Portland Blastfurnace Cement has been issued by the British Standards Institution. This relates to cement containing not less than 65 per cent. of slag and not less than 35 per cent. of Portland cement clinker.

The principal alterations in this specification (known as B.S.146, 1941) compared with the last revision issued in 1932 are in the sections referring to Portland cement, which have been altered to bring them into line with the latest British Standard Specification for Portland Cement (B.S.12) issued earlier this year. A compression test is also now permitted as an alternative to the tensile test. The coarser sieve in the test for fineness of cement is omitted as also is the optional tensile test of neat cement.

Effect of Storage on Portland Cement.

TESTS made to determine the effect of storage under warehouse conditions of an ordinary Portland cement, a rapid hardening Portland cement, and a high-silica blended cement have been made by Mr. K. Watanabe, of the Ube Cement Co., and described in a paper presented to the Association of Japanese Portland Cement Engineers. The chemical analyses of these samples and the calculated content of their chief compounds are given in *Table I.*

Part of each kind of cement in paper sacks and another portion of each type in air-tight tin containers were stored in a room where moisture and temperature were kept under control. The samples were tested after storage periods of 1 and 2 weeks, and 1, 3, 6, and 9 months.

First the uppermost sack of each stack and its corresponding sample in the sealed tin cans were tested. For each succeeding test the uppermost samples remaining were used.

Table II shows the effects on soundness, fineness and setting time for each of the three types of cement. The investigation included also a study of the effects

TABLE I.—COMPOSITION OF CEMENTS.

Kind	Ig. loss	SiO_2	Insol. matter	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	3CS	2CS	3CA	4CAF	Free lime
Normal	0.84	22.84	(0.32)	5.89	2.95	63.72	1.88	1.23	38.14	35.86	10.60	8.97	0.65
Rapid hardening.	0.70	21.88	(0.28)	5.60	3.00	65.02	1.91	1.42	50.14	24.17	9.75	9.12	1.05
High silicious mixed cement	2.08	31.78		6.60	2.80	53.97	1.77	1.37					

on tensile, flexural, and compressive strength, on slump and flow, and on loss by ignition. The room temperature during the tests ranged from 20·5 to 30·5 deg. C. The humidity ranged from 60 to 90·9 per cent. The water temperature ranged from 17 to 26 deg. C.

The normal Portland cement and the rapid-hardening cement had definite increases in setting times for two weeks' storage, after which the setting time of these two cements started to decrease; but even at the 9-month period the setting time was still longer than that of the sealed samples. The high-silica blended cement had a fairly consistent increase in setting time, with a very definite increase at 9 months.

The workability, as measured by the slump test and the flow test, decreased after one month of storage for the normal Portland and the rapid-hardening cement. The high-silica blended cement showed a consistent increase in workability as time progressed. Changes in the workability of the mixtures seemed to be indicated more definitely by means of the slump test than by the flow test.

The effect of storage on the compressive strength was more marked than the effects on tensile or flexural strength. For illustration, the mean compressive strength of normal Portland cement at the 9-month period was only

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Chemical analysis (Argillaceous and calcareous materials, coal and coke, gypsum, cement).

Testing of cement (Setting time, consistency, fineness, soundness, tensile strength).

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62 per cent. of the strength indicated by the corresponding sealed sample. For the same cement the tensile strength at the 9-month test was 96 per cent. of the corresponding sealed sample, while the average flexural strength at 9 months was 73 per cent. of that of the sealed sample.

Comparative strength tests were made on specimens made of 1 : 3 mortar, 1 : 2 mortar, and on 1 : 2 : 4 concrete. Test results indicated that the 1 : 3

TABLE II.—EFFECT OF STORAGE ON SOUNDNESS, FINENESS AND SETTING TIME.

Kind		Period of storage	Soundness (Boiling)	Fineness residue on 4900 sieve	Setting time				
					Temp. °C	Am. of water %	Initial h-m	Final h-m	Moisture %
Normal cement	measured	0	OK	3.5	21.0	26.0	2-46	3-42	70
	Percentage of the stored cement in sack to that in sealed container	1 week				100	68	76	
		2 weeks				102	160	142	
		1 month				100	157	142	
		3 months				98	116	114	
		6 months				104	112	109	
		9 months				102	117	103	
Rapid hardening cement	measured	0	OK	1.0	21.0	26.0	2-0	2-55	70
	Percentage of the stored cement in sack to that in sealed container	1 week				98	103	101	
		2 weeks				102	162	134	
		1 month				100	126	116	
		3 months				104	100	102	
		6 months				107	117	112	
		9 months				101	112	109	
High silicious mixed cement	measured	0	OK	2.5	21.0	30.0	2-49	3-49	70
	Percentage of the stored cement in sack to that in sealed container	1 week				102	113	101	
		2 weeks				100	115	112	
		1 month				100	106	105	
		3 months				101	101	100	
		6 months				105	124	114	
		9 months				100	150	158	

mortar suffered less from storage than the 1 : 2 mortar and the 1 : 2 : 4 concrete, but this difference was not great, and no significance can be attached to it.

After six months' storage the loss in compressive strength ranged from 30 to 40 per cent. on specimens tested at three days, but only from 20 to 30 per cent. on specimens tested at three months.

Setting time was increased to the most marked extent at two weeks and one month, after which this condition seemed to become slowly corrected, the setting time being gradually reduced in length, although at the 9-month period it was still somewhat longer than that of the corresponding sealed sample.

The rapid-hardening cement showed the best resistance to the effect of storage and the high-silica blended cement offered the least resistance; the difference was not great. It was also found that the strength of high-silica blended cement began to decrease at an earlier period than was the case with the other two kinds of cement.

Proposed Reclamation of Lime from Water-Softening Plant.

A FEATURE of a new water-softening and filtration plant of the Metropolitan Water District of Southern California is the reservation of a site for plant for the reclamation of the lime used in softening the water. The present plant has a capacity of 100 million gallons a day, and the average analysis of the water, in parts per million, is as follows: 81 calcium, 25 magnesium, 87 sodium and potassium, 182 bicarbonate, 243 sulphate, 60 chloride, 2·8 nitrate, 0·25 iron, 19 silica, 0·3 fluorine, 0·15 boron, 609 total dissolved solids. The hardness at $CaCO_3$ is 305, of which 145 is carbonate and 160 is non-carbonate. Free carbon dioxide is 6 parts per million, and the hydrogen-ion concentration (pH) is 8·0. The calcium-bicarbonate ($CaCO_3$) hardness of the water is reduced by adding lime or dissolved limestone to the water, resulting in the deposit of insoluble limestone ($2CaCO_3$) as a sludge on the bottom of the clarifier, the reaction being $CaCO_3{\cdot}CO_2 + CaO = C2aCO_3$.

All the water is lime-softened to fewer than 200 parts per million. About one-half of the lime-softened water is zeolite—softened to zero hardness. The combination of the zeolite-softened water with the lime-softened water, on leaving the treatment plant, gives a final hardness of about 85 parts per million..

In the proposed reclamation plant the $CaCO_3$ sludge obtained from the clarifiers will be partially dewatered by a rotary vacuum filter, a continuous centrifuge, or other machine. The cake obtained in this process will be passed to the calcining furnace which may be of the multiple-hearth, rotary, or other type. After the cake has been burned in the furnace it will be classified if necessary to eliminate undesirable grits, and then slaked. The slaked lime will be delivered to the lime-feeding equipment above the mixing channel of the water plant. Carbon dioxide will be obtained as a by-product and can be used for re-carbonation of the water.

This simple procedure could be adopted if there were no, or little, magnesium in the water. But since the water contains 25 parts per million of magnesium as Mg, the repeated reclamation of the calcium carbonate would increase the percentage of magnesium eventually to such an extent that the lime would become unsuitable for softening. The magnesium oxide in this lime would merely be an inert material to be handled repeatedly and in increasing volume. In the procedure actually to be followed the building up of magnesia in the lime obtained from the calcium carbonate must be eliminated or kept within limits assuring desirable operation (see *Fig.* 1).

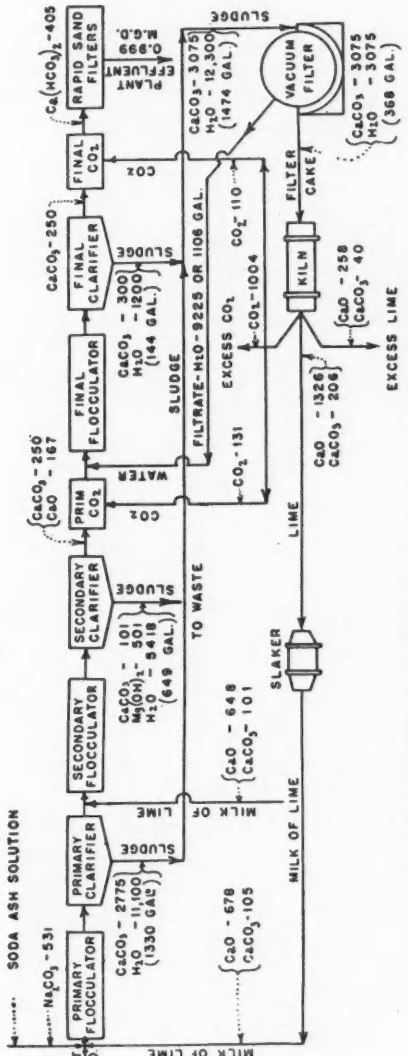

Fig. 1.—Flow Sheet of Reclamation of Lime at Water-softening Plant.

Considering that the water has a total hardness of 305 parts per million and a carbonate hardness of 145 parts per million, and that the treated water will have a total hardness of 125 parts per million, a flow of 100 million gallons a day through the plant results in a theoretical excess production of about 25,800 lb. of CaO, this being essentially free of magnesia. This quantity can be obtained by using the Hoover process of lime reclamation as applied to the softening of water. It is anticipated that lime produced in excess of that needed in the plant can be sold.

The flow-sheet in *Fig.* 1 has been worked out on the basis of handling one million gallons of water per day. It has been assumed that the lime has a solubility of 1·31 gr. of CaO per litre. Just sufficient lime is added in the first stage to precipitate all the calcium present in the bicarbonate form, and the additional lime necessary to precipitate the magnesium is added in the second stage. In the third stage essentially pure calcium carbonate is precipitated in the final clarifier by adding carbon dioxide to the caustic effluent from the secondary stage in which the magnesium is eliminated. The sludge obtained from the primary and final clarifiers or stages is used in reburning, but the sludge obtained from the secondary stage or clarifiers is wasted as it contains the magnesium.

In this flow-sheet all quantities are in pounds on the basis of an average analysis of water of 300 parts per million total hardness and 145 parts per million carbonate hardness, softened to a total hardness of 125 parts per million. The effluent from the rapid sand filters is divided, part flowing through the zeolite softeners and the remainder flowing directly to the plant outfall to join the zeolite-softened water.

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